

Guilford County Emergency Services P.O. Box 18807 Greensboro, N.C. 27419

April 1, 1993

ATTN: Kellly Gage

DAACO, INC.

RE: Additional Information on the Closure at Melvin's Service Station

Kelly,

I'm sorry this closure was so mixed up and incomplete. These tanks were removed during July of 1989. The information I found in these files were based on closure requirements for that time period. Some of the differences were, composite sampling for tanks with a threshold of 100 ppm for contamination. No chain of custody was required then either. I have included all the information on file to try and establish some continuity. The four gas tanks were removed July 13, 1989. Samples were taken and delivered to Guilford Laboratories that same afternoon, (invoice #DAC90713). DAACO had acquired an O.V.A. meter at that time and it was used to scan the excavation. One section did exhibit reading above normal. This was attributed to organic trash in the backfill at that point. The laboratory analysis returned with values above the threshold in the same section and soil was removed until the O.V.A. meter no longer indicated any contaminates. Another sample was tested from that section of the gasoline excavation along with a sample taken from the diesel excavation. The diesel tank removal and remediation were both performed July 31, 1989. Both samples returned within acceptable levels, (invoice #DAC90731). The soil retrieved from the gas excavation, (appox. 23 cubic yds/two tandem loads) was held on site. When discussing this site with you in January, I suggested sampling this soil to determine if any hydrocarbons were still present. The sample was tested for EPA-5030 leaded and unleaded, (Burlington Research #93-20-090). The results were in the acceptable range.

I hope this summary along with the supporting information will be sufficient to complete the closure. Again I apologize for any inconvenience this may have caused you.

Thank you,

DAACO, INC.

James Harris

BR-17 11-91 White—ORIGINAL - CLIENT REPORT	Method of Shipment:	FOR LAB USE ONLY Received in Lab FROM:	Method of Shipment:	Shipped by: (Signature)		FOR CLIENT USE:						Tang & Just			SAMPLE		(919) 584-5564 Fax (919) 584-5564, Ext. 202	Research	Burlington	n
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Analytical Services • Aquatic Bioassay Testing • Aquatic Toxicity Reduction Evaluations AATCC Testing Services • NPDES Testing • Reporting & Data Handling Services PMN Aquatic Bioassay Evaluations

Post Office Box 2481 • 615 Huffman Mill Road • (919) 584-5564 • Burlington, NC 27216-2481

ANALYTICAL REPORT

CUSTOMER:

WORK ORDER #:

93-02-090-01

FACILITY:

COLLECTED:

01/25/93

REPORT TO:

DAACO, INC. 5212 W. Market St. James 'Chip' Harris

RECEIVED:

SAMPLE:

Tom Sawyer

Back Ground 1 Grab 1/25/93

REPORTED:

02/05/93 02/11/93

PARAMETER	METHOD	STARTED	ANALYZED	RESULT
TPH-I, Soils	EPA 5030/GC	02/08/93	02/08/93	Attached

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AATCC Testing Services • NPDES Testing • Reporting & Data Handling Services
PMN Aquatic Bioassay Evaluations

Post Office Box 2481 • 615 Huffman Mill Road • (919) 584-5564 • Burlington, NC 27216-2481

EPA METHOD 5030 WITH CALIFORNIA GC METHOD TOTAL PETROLEUM HYDROCARBONS - TYPE I, IN SOILS

WORK ORDER #: 93-02-090-01

TPH: ≤ 10.0 mg/kg (ppm)

TPH Standard used: Leaded Gasoline

% Recovery of Standard: 130

% Solids: <u>77</u>

Analytical Services • Aquatic Bioassay Testing • Aquatic Toxicity Reduction Evaluations

AATCC Testing Services • NPDES Testing • Reporting & Data Handling Services

PMN Aquatic Bioassay Evaluations

Post Office Box 2481 • 615 Huffman Mill Road • (919) 584-5564 • Burlington, NC 27216-2481

EPA METHOD 5030 WITH CALIFORNIA GC METHOD TOTAL PETROLEUM HYDROCARBONS - TYPE I, IN SOILS

WORK ORDER #: 93-02-090-01

TPH: ≤ 10.0 mg/kg (ppm)

TPH Standard used: <u>Unleaded Gasoline</u>

% Recovery of Standard: 130

% Solids: <u>77</u>

METHOD 3540A

SOXHLET EXTRACTION

1.0 SCOPE AND APPLICATION

- 1.1 Method 3540 is a procedure for extracting nonvolatile and semi-volatile organic compounds from solids such as soils, sludges, and wastes. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.
- 1.2 This method is applicable to the isolation and concentration of water insoluble and slightly water soluble organics in preparation for a variety of chromatographic procedures.

2.0 SUMMARY OF METHOD

2.1 The solid sample is mixed with anhydrous sodium sulfate, placed in an extraction thimble or between two plugs of glass wool, and extracted using an appropriate solvent in a Soxhlet extractor. The extract is then dried, concentrated, and, as necessary, exchanged into a solvent compatible with the cleanup or determinative step being employed.

3.0 INTERFERENCES

3.1 Refer to Method 3500.

4.0 APPARATUS AND MATERIALS

- 4.1 Soxhlet extractor 40 mm ID, with 500 mL round bottom flask.
- 4.2 Drying column 20 mm ID Pyrex chromatographic column with Pyrex glass wool at bottom.

NOTE: Fritted glass discs are difficult to decontaminate after highly contaminated extracts have been passed through. Columns without frits may be purchased. Use a small pad of Pyrex glass wool to retain the adsorbent. Prewash the glass wool pad with 50 mL of acetone followed by 50 mL of elution solvent prior to packing the column with adsorbent.

4.3 Kuderna-Danish (K-D) apparatus

- 4.3.1 Concentrator tube 10 mL, graduated (Kontes K-570050-1025 or equivalent). A ground glass stopper is used to prevent evaporation of extracts.
- 4.3.2 Evaporation flask 500 mL (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs, clamps, or equivalent.

- 4.3.3 Snyder column Three ball macro (Kontes K-503000-0121 or equivalent).
- 4.3.4 Snyder column Two ball micro (Kontes K-569001-0219 or equivalent).
 - 4.3.5 Springs 1/2 inch (Kontes K-662750 or equivalent).
- 4.4 Boiling chips Solvent extracted, approximately 10/40 mesh (silicon carbide or equivalent).
- 4.5 Water bath Heated, with concentric ring cover, capable of temperature control (\pm 5°C). The bath should be used in a hood.
 - 4.6 Vials Glass, 2 mL capacity, with Teflon lined screw or crimp top.
 - 4.7 Glass or paper thimble or glass wool Contaminant free.
 - 4.8 Heating mantle Rheostat controlled.
 - 4.9 Disposable glass pasteur pipet and bulb.
 - 4.10 Apparatus for determining percent dry weight.
 - 4.10.1 Oven Drying.
 - 4.10.2 Desiccator.
 - 4.10.3 Crucibles Porcelain or disposable aluminum.
 - 4.11 Apparatus for grinding
 - 4.12 Analytical balance 0.0001 q.

5.0 REAGENTS

- 5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.
- 5.2 Organic-free reagent water. All references to water in this method refer to organic-free reagent water, as defined in Chapter One.
- 5.3 Sodium sulfate (granular, anhydrous), Na_2SO_4 . Purify by heating at 400°C for 4 hours in a shallow tray, or by precleaning the sodium sulfate with methylene chloride. If the sodium sulfate is precleaned with methylene chloride, a method blank must be analyzed, demonstrating that there is no interference from the sodium sulfate.

5.4 Extraction solvents

- 5.4.1 Soil/sediment and aqueous sludge samples shall be extracted using either of the following solvent systems:
 - 5.4.1.1 Toluene/Methanol (10:1) (v/v), $C_6H_5CH_3/CH_3OH$. Pesticide quality or equivalent.
 - 5.4.1.2 Acetone/Hexane (1:1) (v/v), $CH_3COCH_3/CH_3(CH_2)_4CH_3$. Pesticide quality or equivalent.
 - 5.4.2 Other samples shall be extracted using the following:
 - 5.4.2.1 Methylene chloride, CH_2Cl_2 . Pesticide quality or equivalent.

5.5 Exchange solvents

- 5.5.1 Hexane, C_6H_{14} . Pesticide quality or equivalent.
- 5.5.2 2-Propanol, $(CH_3)_2CHOH$. Pesticide quality or equivalent.
- 5.5.3 Cyclohexane, C_6H_{12} . Pesticide quality or equivalent.
- 5.5.4 Acetonitrile, CH₃CN. Pesticide quality or equivalent.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analysis, Section 4.1.

7.0 PROCEDURE

7.1 Sample Handling

- 7.1.1 Sediment/soil samples Decant and discard any water layer on a sediment sample. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks.
- 7.1.2 Waste samples Samples consisting of multiphases must be prepared by the phase separation method in Chapter Two before extraction. This procedure is for solids only.
- 7.1.3 Dry waste samples amenable to grinding Grind or otherwise subdivide the waste so that it either passes through a 1 mm sieve or can be extruded through a 1 mm hole. Introduce sufficient sample into the grinding apparatus to yield at least 10 g after grinding.
- 7.2 Determination of sample % dry weight In certain cases, sample results are desired based on dry weight basis. When such data is desired, a portion of sample for this determination should be weighed out at the same time as the portion used for analytical determination.

<u>WARNING</u>: The drying oven should be contained in a hood or vented.

Significant laboratory contamination may result from a heavily contaminated hazardous waste sample.

7.2.1 Immediately after weighing the sample for extraction, weigh 5-10 g of the sample into a tared crucible. Determine the % dry weight of the sample by drying overnight at 105° C. Allow to cool in a desiccator before weighing:

% dry weight = $g ext{ of dry sample} ext{ x 100}$ g of sample

- 7.3 Blend 10 g of the solid sample with 10 g of anhydrous sodium sulfate and place in an extraction thimble. The extraction thimble must drain freely for the duration of the extraction period. A glass wool plug above and below the sample in the Soxhlet extractor is an acceptable alternative for the thimble. Add 1.0 mL of the surrogate standard spiking solution onto the sample (see Method 3500 for details on the surrogate standard and matrix spiking solutions). For the sample in each analytical batch selected for spiking, add 1.0 mL of the matrix spiking standard. For base/neutral-acid analysis, the amount added of the surrogates and matrix spiking compounds should result in a final concentration of 100 ng/ μ L of each base/neutral analyte and 200 ng/ μ L of each acid analyte in the extract to be analyzed (assuming a 1 μ L injection). If Method 3640, Gel Permeation Chromatography Cleanup, is to be used, add twice the volume of surrogates and matrix spiking compounds since half the extract is lost due to loading of the GPC column.
- 7.4 Place approximately 300 mL of the extraction solvent (Section 5.4) into a 500 mL round bottom flask containing one or two clean boiling chips. Attach the flask to the extractor and extract the sample for 16-24 hours at 4-6 cycles/hr.
 - 7.5 Allow the extract to cool after the extraction is complete.
- 7.6 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10 mL concentrator tube to a 500 mL evaporation flask.
- 7.7 Dry the extract by passing it through a drying column containing about 10 cm of anhydrous sodium sulfate. Collect the dried extract in a K-D concentrator. Wash the extractor flask and sodium sulfate column with 100 to 125 mL of extraction solvent to complete the quantitative transfer.
- 7.8 Add one or two clean boiling chips to the flask and attach a three ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (15-20°C above the boiling point of the solvent) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-20 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 1-2 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

- 7.9 If a solvent exchange is required (as indicated in Table 1), momentarily remove the Snyder column, add approximately 50 mL of the exchange solvent and a new boiling chip, and reattach the Snyder column. Concentrate the extract as described in Section 7.8, raising the temperature of the water bath, if necessary, to maintain proper distillation. When the apparent volume again reaches 1-2 mL, remove the K-D apparatus from the water batch and allow it to drain and cool for at least 10 minutes.
- 7.10 Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1-2 mL of methylene chloride or exchange solvent. If sulfur crystals are a problem, proceed to Method 3660 for cleanup. The extract may be further concentrated by using the techniques described in Section 7.11 or adjusted to 10.0 mL with the solvent last used.
- 7.11 If further concentration is indicated in Table 1, either micro Snyder column technique (7.11.1) or nitrogen blowdown technique (7.11.2) is used to adjust the extract to the final volume required.

7.11.1 Micro Snyder Column Technique

7.11.1.1 Add another one or two clean boiling chips to the concentrator tube and attach a two ball micro Snyder column. Prewet the column by adding about 0.5 mL of methylene chloride or exchange solvent to the top of the column. Place the K-D apparatus in a hot water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 5-10 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 0.5 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. Remove the Snyder column and rinse the flask and its lower joints with about 0.2 mL of solvent and add to the concentrator tube. Adjust the final volume to 1.0-2.0 mL, as indicated in Table 1, with solvent.

7.11.2 Nitrogen Blowdown Technique

7.11.2.1 Place the concentrator tube in a warm water bath (approximately 35°C) and evaporate the solvent volume to the required level using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon).

<u>CAUTION:</u> Do not use plasticized tubing between the carboπ trap and the sample.

7.11.2.2 The internal wall of the tube must be rinsed down several times with the appropriate solvent during the operation. During evaporation, the solvent level in the tube must be positioned to prevent water from condensing into the sample (i.e., the solvent level should be below the level of the water bath). Under normal operating conditions, the extract should not be allowed to become dry.

<u>CAUTION</u>: When the volume of solvent is reduced below 1 mL, semivolatile analytes may be lost.

7.12 The extracts obtained may now be analyzed for the target analytes using the appropriate organic technique(s) (see Section 4.3 of this Chapter). If analysis of the extract will not be performed immediately, stopper the concentrator tube and store in a refrigerator. If the extract will be stored longer than 2 days, it should be transferred to a vial with a Teflon lined screw cap or crimp top, and labeled appropriately.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures and Method 3500 for extraction and sample preparation procedures.

9.0 METHOD PERFORMANCE

9.1 Refer to the determinative methods for performance data.

10.0 REFERENCES

 U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.

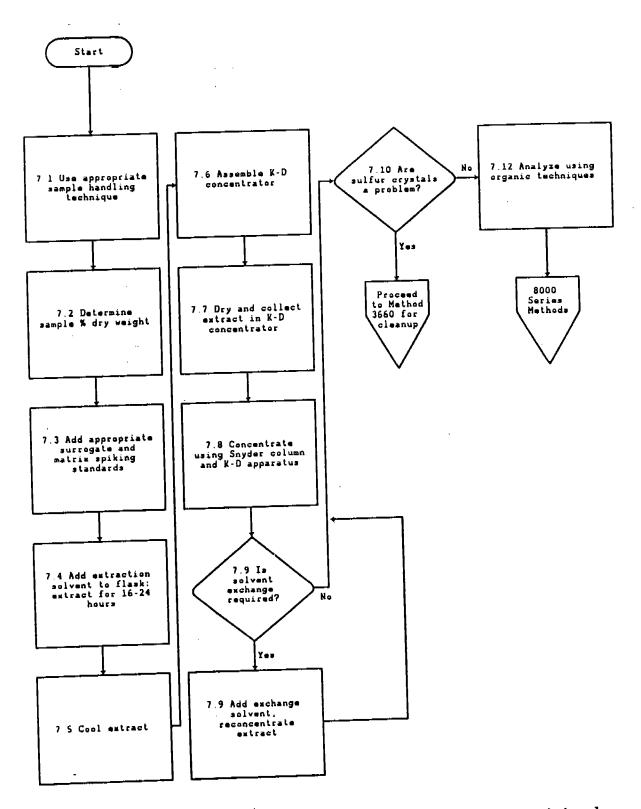
TABLE 1. SPECIFIC EXTRACTION CONDITIONS FOR VARIOUS DETERMINATIVE METHODS

Determinative method	Extraction pH	Exchange solvent required for analysis	Exchange solvent required for cleanup	Volume of extract required for cleanup (mL)	Final extract volume for analysis (mL)
8040° 8060 8070 8080 8100 8110 8141 8250° 8310	as received	2-propanol hexane hexane hexane hexane hexane hexane hexane hexane hexane none none acetonitrile	hexane hexane hexane cyclohexane hexane hexane hexane hexane	1.0 2.0 2.0 2.0 2.0 10.0	1.0, 10.0° 10.0 10.0 1.0 10.0 10.0 10.0 1.0

^a To obtain separate acid and base/neutral extracts, Method 3650 should be performed following concentration of the extract to 10.0 mL.

b Phenols may be analyzed by Method 8040, using a 1.0 mL 2-propanol extract by GC/FID. Method 8040 also contains an optical derivatization procedure for phenols which results in a 10 mL hexane extract to be analyzed by GC/ECD.

^c The specificity of GC/MS may make cleanup of the extracts unnecessary. Refer to Method 3600 for guidance on the cleanup procedures available if required.



Revision 1 November 1990

August 8, 1989

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REPORT OF ANALYSIS

JOB #:

DAC90731

CUSTOMER:

Daaco Inc

ATTN: Dave Andrews

SAMPLE:

3597 SOIL TST #1

3598 SOIL TST #2

RESULTS

3597

3598

Total Petroleum Hydrocarbons (ppm)

Guilford Laboratories, Inc. JA Kayburn JA

Sauger-Bero

August 8, 1989

REPORT OF ANALYSIS

JOB #1

CUSTOMER:

Deaco Inc

ATTN: Dave Andre

SAMPLE:

3597 SOIL TST #1

3598 SOIL TST #2

RESULTS

3597

3598

Total Petroleum Hydrocarbons (ppm)

5

Guilford Laboratories, Inc. 1. Kaybur IX

J. A. Rayburn

Safeway Tank Disposal, Inc.

Page / of /

CERTIFICATE OF TANK DISPOSAL

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Customer	0.				Date _	7-31-19
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Tank Disposal Number	Size	Weight	Product	Residue Amount		Origin
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			0		0077	<u> </u>
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			Total Residue	2		
						and Disposal of Used cordance with U.S. EPA
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				_	SAFEWAY TANK DIS	

Safeway Tank Disposal, Inc.

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Sawur RECEIVING REPORT

From: DAACO					Received by: DAN STOWE SAFEWAY TANK DISPOSAL, INC.							
			_	Transported by:								
Tank Disposal Number Size Weight		Weight	Prodi	uct	Date Received			Origin				
1253	550		DIE	SEL	7-31		SAWWER S ASHBORD	ST GRE	ENSBORO			
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Safeway Tank Disposal, Inc. accepts the liability for the tank(s) and contents on this report. The tank(s) and contents must be a petroleum product. If at any time the tanks are found to contain any product other than a petroleum product SAFEWAY TANK DISPOSAL, INC. has the right to refuse disposal or negotiate a price for disposal. Customer will be liable for any clean-up or other cost resulting from contamination by a substance other than a petroleum product.

Safeway Tank Disposal, Inc. agrees to dispose of petroleum tanks and contents in accordance with local, state, and federal regulation. Certificate of Disposal to follow.

SAFEWAY TANK DISPOSAL, INC.

Safeway Tank Disposal, Inc.

Page | of |

RECEIVING REPORT

From:	rom: DAACO				Received by: DAN STOWE SAFEWAY TANK DISPOSAL, INC.							
-			_	Tra	nsported by:	DAACO						
Tank Disposal Number	Size	Weight	Produ	uct	Date Received	Origin						
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1218	2000		GA	S	7-14-89							
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Safeway Tank Disposal, Inc. accepts the liability for the tank(s) and contents on this report. The tank(s) and contents must be a petroleum product. If at any time the tanks are found to contain any product other than a petroleum product SAFEWAY TANK DISPOSAL, INC. has the right to refuse disposal or negotiate a price for disposal. Customer will be liable for any clean-up or other cost resulting from contamination by a substance other than a petroleum product.

Safeway Tank Disposal, Inc. agrees to dispose of petroleum tanks and contents in accordance with local, state, and federal regulation. Certificate of Disposal to follow.

SAFEWAY TANK DISPOSAL, INC.

July 25, 1989

REPORT OF ANALYSIS

JOB #:

DAC90713

CUSTOMER: Daaco Inc

P.O. #:

Sawyer

SAMPLE:

3440, SOIL TANK #1

3441

SOIL TANK #2

3442 SOIL TANK #3

3443 SOIL TANK #4

RESULTS		<u> 3440</u>	<u>3441</u>	<u>3442</u>	3443
lotal Petroleum Hydrocarbons	(ppm)	2650	67	(10*	(10

Sample Contains Non-Petroleum Hydrocarbons.

Guilford Laboratories, Inc.

J. A. Rayburn

July 25, 1989

REPORT OF ANALYSIS

JOB #:

DAC90713

CUSTOMER: Daaco Inc

P.O. #:

Sawyer

SAMPLE:

SOIL TANK #1 # 3440

3441

SOIL TANK #2

3442

SUIL TANK #3 # 3443 SOIL TANK #4

RESULTS

3440

Total Petroleum Hydrocarbons (ppm)

67

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Sample Contains Non-Petroleum Hydrocarbons.

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Builford Laboratories, Inc. 1. Kazbum DE

J. A. Rayburn